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SYSTEM OPTIMIZATION OF THE GLOW DISCHARGE OPTICAL SPECTROSCOPY --ETC (11)
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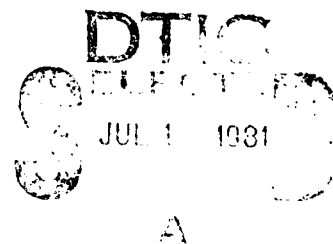
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SYSTEM OPTIMIZATION OF THE GLOW
DISCHARGE OPTICAL SPECTROSCOPY
TECHNIQUE USED FOR IMPURITY PROFILING
OF ION IMPLANTED GALLIUM ARSENIDE

THESIS

AFIT/GEO/EE/80D-1

Paul J. Apuzzo
Capt USAF



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SPECTROSCOPY TECHNIQUE USED FOR IMPURITY
PROFILING OF ION IMPLANTED
GALLIUM ARSENIDE .

THESIS

Presented to the Faculty of the School of Engineering
of the Air Force Institute of Technology
Air University
In Partial Fulfillment of the
Requirements for the Degree of
Master of Science

by
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Preface

Glow discharge optical spectroscopy (GDOS) is a technique used for measuring impurity concentration profiles in semiconductors, specifically annealed and unannealed ion implanted gallium arsenide (GaAs). Methods to improve the sensitivity of the GDOS system have been investigated. These efforts indicate the desirability of continued investigation and development of the system.

I would like to thank the numerous Avionics Laboratory personnel for their valuable guidance and assistance. These include Dr. Y. S. Park for providing me with this thesis topic and J. Ray without whose excellent glass work this thesis would not have been possible. Also, thanks to C. Geesner, J. Ehret, G. Johnson, and Capt W. Theis for their valuable assistance. Thanks to Professor S. Yun of Ohio State University for his help in teaching me to operate the GDOS apparatus and to the AFIT Fabrication Division for providing metal work essential to this study.

I would also like to thank Major Borkey, my thesis advisor, for allowing me to choose my own path in this experimental study. Special thanks go to my wife for her patience and aid in the preparation of this manuscript and to my daughter for putting up with my absence during the past months.

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Abstract

The glow discharge optical spectroscopy (GDOS) technique was investigated to determine if the sensitivity of the system could be improved. GDOS was applied to determine impurity concentration profiles of annealed and unannealed ion implanted GaAs samples. Modifications were made to the sputtering chamber and the light collection systems which resulted in a 3.7 times increase in system sensitivity. Comparisons were made to previous studies by sputtering GaAs samples implanted with Ge at energies of 90 keV and 120 keV and fluences of $5 \times 10^{14}/\text{cm}^2$ and $10^{15}/\text{cm}^2$. The implanted samples were sputtered in a low pressure argon gas discharge. Intensity of a strong emission line, characteristic of the implanted impurity, was monitored as a function of time. Profiles of Ge implanted GaAs were measured and analyzed.

*s. x. 10 to the 14th power/cm²
and 10 to the 15th power/cm².*

SYSTEM OPTIMIZATION OF THE GLOW DISCHARGE OPTICAL
SPECTROSCOPY TECHNIQUE USED FOR IMPURITY
PROFILING OF ION IMPLANTED
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I. Introduction

Background

The Air Force is interested in using GaAs, a III-IV semiconductor, for a variety of devices (Refs 13, 21, 22). GaAs exhibits higher electron mobilities and band gap than Si or Ge. The Electronics Research Branch of the Microelectronics Division, Avionics Laboratory, Air Force Wright Aeronautical Laboratories (AFWAL/AADR) is currently conducting research to develop the basic technology needed to produce devices on GaAs substrates.

Devices will exhibit different characteristics depending on the type and amount of impurity atoms introduced into the GaAs substrate. The method most often used for doping GaAs is the ion implantation technique. This technique produces the desired characteristic of precisely controlled and reproducible impurity profiles. Unfortunately, ion implantation damages the crystal lattice, and the damage must be removed by annealing the crystal. Implanted impurities will diffuse during this annealing, altering the impurity profile and thus, the electrical

characteristics of devices made from such materials. Precise knowledge of the annealing effects and the revised dopant distribution is needed to perfect the technology necessary to produce GaAs devices.

Several methods are currently used for measuring impurity profiles in GaAs. Secondary ion mass spectroscopy (SIMS) (Ref 10), Auger electron spectroscopy (AES) (Ref 15), and glow discharge optical spectroscopy (GDOS) (Ref 7) have been successfully employed in determining the total atomic distribution of implants. Hall-effect (Ref 24), capacitance-voltage (Ref 14), and luminescence measurements (Ref 25) are also used to obtain the distribution of electrically or optically active impurity atoms. Selection of the appropriate technique of those previously mentioned is governed by the experimental limitations, sensitivity of detection required, and electrical and optical activity of the implants.

Objectives

The purpose of this thesis was to investigate methods to improve the sensitivity of the present GDOS apparatus. Chamber modifications and optical collection improvements were studied in an effort to enhance the light collection of the system. Once an acceptable sensitivity improvement was achieved, GaAs:Cr was to be analyzed to determine the effect of several annealing strategies on the impurity profile of Cr.

In GDOS, the implanted substrate is dc sputtered in a pyrex vacuum chamber which is backfilled with Ar. The sputtering occurs as the ionized Ar atoms strike the surface of the sample placed on the cathode. The atoms emitted from the substrate are collisionally excited in the plasma and emit a characteristic luminescence. With the use of pure elements such as pure Ge, and knowing the substrate sputtering rate, the measured emission intensity may be related to the concentration of impurity ions in the substrate (Ref 7:366).

Results

The majority of the work in this study involved a complete reworking of the electrical, optical, and vacuum components of the system. By optimizing the optical collection system and improving the vacuum integrity, the productivity of the GDOS system was doubled.

The chamber was tilted 30° to enhance the collection of light emitted from the sample. Increased impurity signal was observed, resulting in an increase in system sensitivity by a factor of two. Despite the improvements made, the system noise level was too high to detect Cr in GaAs:Cr. Strong interference was caused by the Cr impurities in the stainless steel anode and tungsten cathode.

A vertical chamber was installed with a flat mirror and lens arrangement to capture light emitted rearward. The result was a 3.7 factor increase in system sensitivity.

Organization

Chapter II contains a discussion of the theory of ion implantation and its use in GaAs device structures. After ion implantation is accomplished, impurity profiling is necessary to check the distribution of impurity atoms within the substrate. This is accomplished by glow discharge optical spectroscopy, which is discussed in Chapter III. To fully understand the GDOS system, the experimental apparatus used in this study is presented in Chapter IV. Optimizing the capabilities of this apparatus is important for achieving the maximum sensitivity of the system. For this reason, system improvements are discussed in Chapter V. Some of the improvements described here were accomplished and others are for future considerations. Now that the system has been explained, the procedures and the calibration techniques used in performing the experiment are presented in Chapter VI. The results of the system improvements and the impurity profiling performed are analyzed in the discussion in Chapter VII. Included in this section are the results obtained by introducing small amounts of He into the chamber. Conclusions drawn from this experiment such as the present and projected system sensitivity are presented in the last chapter of this report.

II. Ion Implantation

Ion implantation has become increasingly important in the fabrication of semiconductor devices. Advantages of ion implantation include the ability to control and reproduce both the number of impurity atoms introduced into the crystal and their depth distribution. In addition, the technique has the potential of introducing any ion into any substrate material. Ion implantation can produce the shallow, precise layers of dopant required in field effect transistors (FET), optical, and transferred electron device (TED) structures. For dopants with very low diffusion coefficients, ion implantation can be used in lieu of conventional diffusion doping. Ion implantation is a low temperature process which will not cause GaAs to dissociate. High temperature diffusion, on the other hand, has limited use on GaAs since it can cause dissociation of the host lattice.

Ion implantation is achieved by bombarding a substrate with high energy ions of uniform mass and energy. This bombardment produces a spacial distribution or doping profile of the incident ions below the substrate surface. However, it also produces a substantial amount of crystal lattice disorder, or damage. The damage can be repaired by annealing the crystal at moderate temperatures in a flowing inert gas atmosphere. Prior to annealing, the substrate

is coated with a thin layer of SiO_2 or Si_3N_4 . This coating has been found effective in preventing dissociation of GaAs, as well as reducing the outdiffusion of the implanted ions during annealing (Ref 20:214).

History

The first attempt at ion implantation into semiconductors was made by Cussins in 1955 (Ref 5:296). He implanted B ions into Ge substrates, and obtained a p-type conductivity. Cussins concluded that the principal effect of the ion bombardment was damage of the crystalline lattice of the Ge.

Many theories to predict the implanted ion distribution in amorphous targets have been developed since that time (Refs 5, 10, 14, 15). The most prominent and most widely accepted theory is the Linhard, Scharff, and Schiott (LSS) range distribution theory developed in 1963 (Ref 11: 1-39). This theory assumes that sputtering of surface atoms is negligible in ion implantation, and that the crystalline target is amorphous or purposely misaligned with respect to any high symmetry directions.

GaAs has a zincblende structure composed of Ga atoms in the (111) face and As atoms in the ($\bar{1}\bar{1}\bar{1}$) face producing interpenetrating face centered cubic (fcc) sub-lattices (Ref 3:262). The widely accepted method of implanting GaAs is to cleave the samples along a (100) plane and purposely misalign the target 8° from the normal for implantation.

Thus, the GaAs target is effectively amorphous and the LSS theory is applicable.

Range Estimates

The total range, R , that an implanted ion will travel before coming to rest in a given substrate is a function of that substrate's nuclear and electronic stopping powers. The projection of this range on the incident ion direction is called the projected range, R_p , which can be determined experimentally (Ref 5:299). The relationship between R_p and R is shown in Figure 1.

Due to the randomness of collisions between incident and target atoms, ions of a given type and energy come to rest in the substrate at different depths, forming a range distribution. This distribution is approximately gaussian in amorphous targets as shown in Figure 2. The range distribution can be found by using the calculated values for the projected ranges, R_p , and their standard deviations, ΔR_p , which have been tabulated for the ions and substrate used in this investigation (Ref 6).

Thus, it has been shown that ion implantation into a GaAs target aligned 8° from the normal during implantation can yield a uniform gaussian distribution of the implanted ions. Since the GaAs target was effectively amorphous during implantation, the LSS theory is applicable for predicting the implanted ion distribution. The resulting gaussian distribution is defined by the parameters R_p

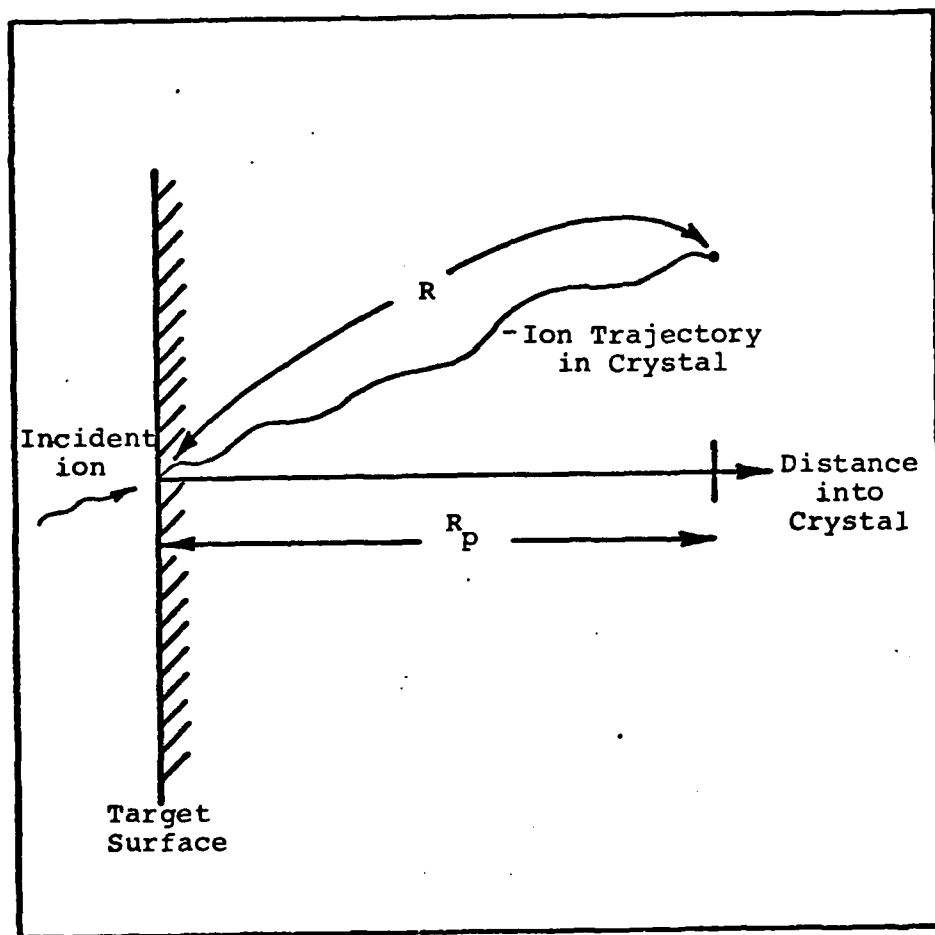


Figure 1. Definition of Range R and Projected Range R_p

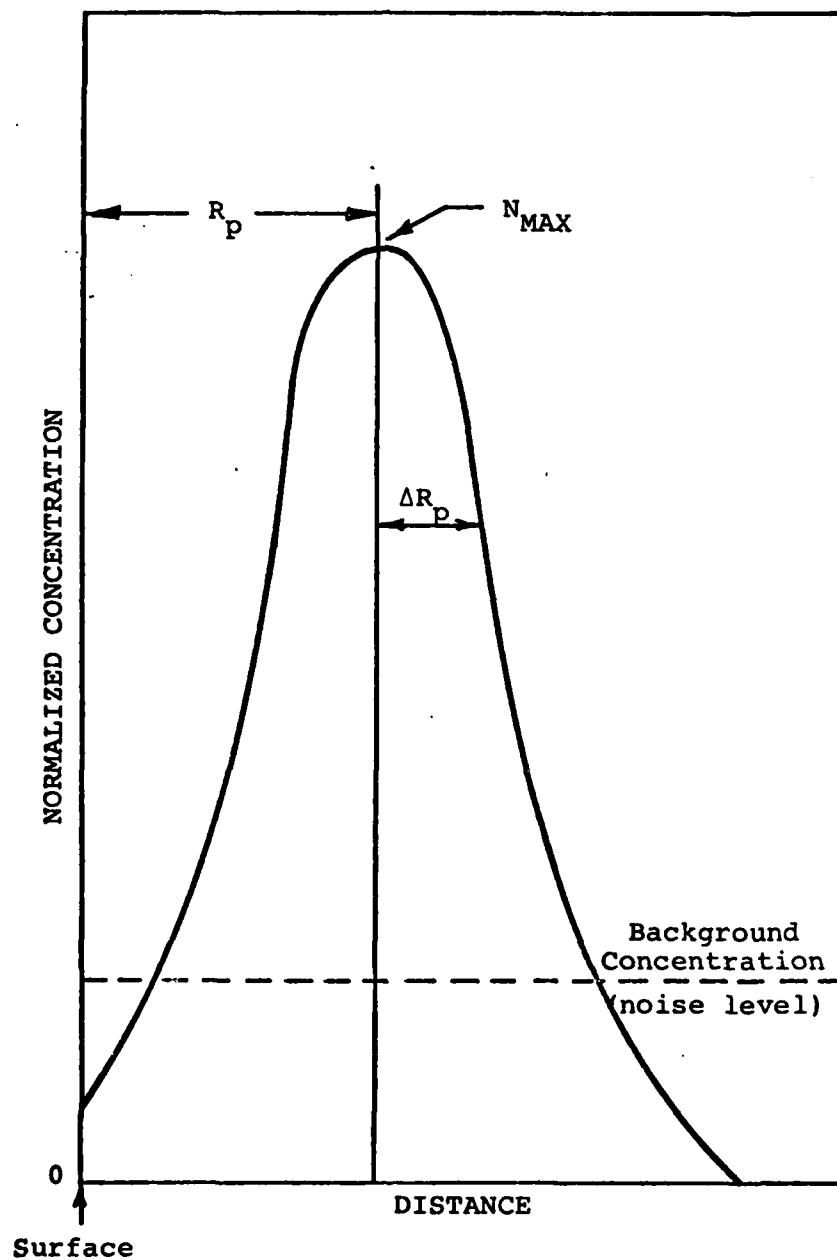


Figure 2. Gaussian Distribution in an Amorphous Target

and $\overline{\Delta R_p}$. One technique for evaluating implanted profiles is glow discharge optical spectroscopy, which is the topic of discussion in Chapter III, GDOS section.

III. Glow Discharge Optical Spectroscopy

Discharge

The electric gas discharge is the phenomena whereby a gas or vapour becomes electrically conducting. The gas becomes ionized and the free electrons move through the gas under the influence of an electric field. This phenomena can be observed in a tube filled with gas to a few millimeters of pressure, while a potential is applied across the electrodes. The result is a glow discharge which consists of a series of alternate luminous and dark zones as shown in Figure 3 (Ref 16:125-140). When the distance between the anode and cathode of a glow discharge is varied, it has been found that the axial length of the positive column varies, but the sizes of the other regions are not affected (Ref 4:191). Also, decreasing the gas pressure increases the size of the negative glow and Faraday dark space regions and causes the positive column to decrease or disappear (Ref 4:190-193). This indicates that the phenomena at and near the cathode are essential to the discharge, and that the positive column merely serves to maintain a conducting path for the current (Ref 1:214).

The discharge itself is produced when gas molecules are ionized and the resulting positive ions are accelerated towards and strike the cathode. Electrons emitted from the cathode are accelerated by the electric field and

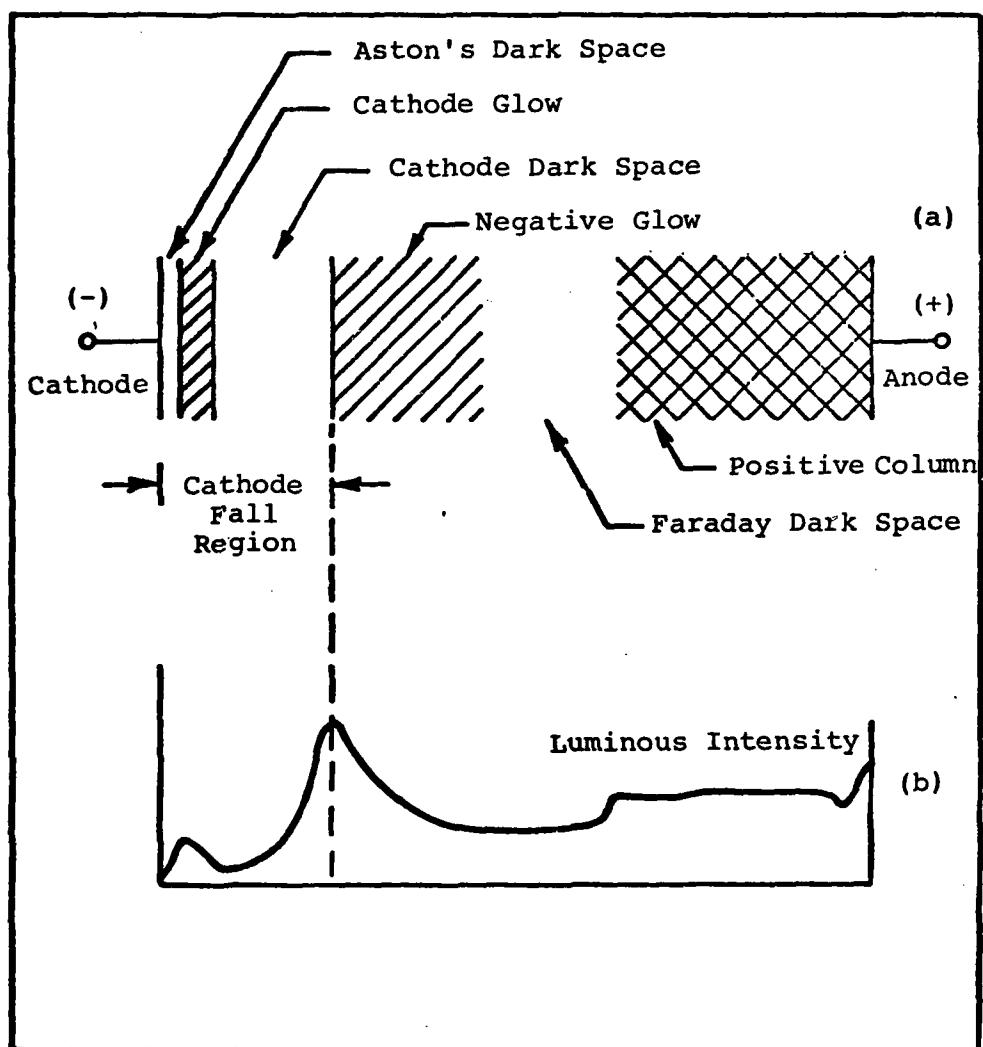


Figure 3. (a) Discharge Structure in a Cathode Region
 (b) Luminous Intensity Distribution
 (From Ref 16:126)

collide with gas molecules as the electrons travel toward the anode. These emitted electrons initially have energies too low to collisionally excite the gas. This accounts for the dark space immediately next to the cathode called Aston's dark space. The luminous region next to Aston's dark space is called the cathode glow region. In this region the ions excited by electrons radiatively decay. The cathode dark space is the region where the gas is ionized by the electrons which have been accelerated by the electric field but have not experienced any collisions. The electrons emitted due to this ionization do not have enough energy to excite the gas, and therefore there is little emission in this region of the discharge (Ref 15: 130).

The electrons liberated in the cathode dark space are accelerated by the electric field and attain sufficient energy to collisionally excite gas molecules and emit light. The light emitted in this region (called the negative glow region) is much more intense than in the cathode glow region. Beyond this part of the discharge, the electrons again lose their energy and cannot excite or ionize the gas. The field in this region is so small that the electrons cannot regain much energy. This produces the area known as the Faraday dark space. The positive column fills the region from the Faraday dark space to the anode sheath. In this region, the electrons accelerate again

and collide producing light. This is the most luminous part of the tube after the negative glow (Ref 16:131).

The discharge regions of interest in GDOS are Aston's dark space, the cathode glow region, and the cathode dark space. These three regions make up the cathode fall region. A normal glow discharge occurs when the current density, dark space, and cathode fall voltage remain constant when the current is raised by several orders of magnitude (Figure 4). The cathode is only partially covered by the glow in this type of discharge.

An abnormal glow discharge occurs when the negative glow region completely covers the cathode. An increase in current produces an increase in cathode fall voltage, and an increase in current density (Figure 4). This results in a decrease in the thickness of the cathode dark space. It is in this abnormal glow discharge that most sputtering is done.

Sputtering

In a glow discharge, the cathode is subjected to bombardment by positive ions. This causes a continual erosion of the cathode surface which is called sputtering. Sputtering yield is defined as the total number of ejected atoms per incident ion. It is a function of the gas pressure, concentration of implanted ions, electric field at the surface of the target, and cathode current density. The amount of sputtering is also dependent on the masses

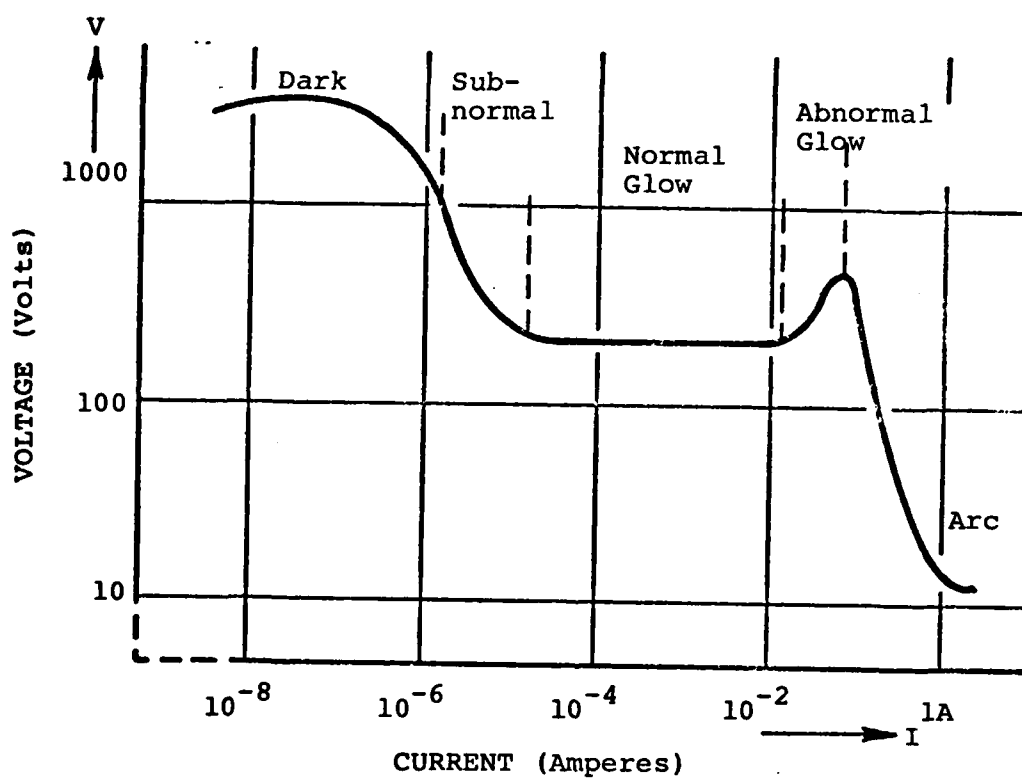


Figure 4. Schematic Characteristics for a Gaseous Discharge (Ref 16:123)

of the ions and the target atoms, the ion energy and the angle of incidence to the face of the target (Ref 20:111-128). Maximum sputtering yield occurs at normal incidence, and sputtering increases for higher ion masses and energies (Ref 19:679). However, it has been empirically noticed that at higher gas pressures there is a reduction in the sputtering yield. In this case, incident positive ions experience more collisions while moving towards the cathode and therefore strike the cathode with less energy. High pressure sputtering may also cause increased contamination of the discharge since ejected atoms may be reflected back toward the cathode. In this case, profiling results will not directly correspond to the amount of impurity atoms in the substrate.

The material sputtered in a glow discharge consists almost entirely of neutral atoms. The momentum of the incident ions is transferred along crystallographic or close packing directions and results in preferential sputtering in certain directions. Studies show that when GaAs is sputtered at normal incidence, the neutral Ga and As atoms are ejected stoichiometrically from the (111) face of the substrate (Ref 2:2956-2960).

The amount of light collected may be enhanced by mixing in small amounts of He (on the order of 1 to 5%) with the Ar sputtering gas. The He ions have a much lower sputtering yield than Ar ions because of their lower atomic mass. However, the degree of Ar ionization in the plasma

has been shown to increase with the He ions added due to the Penning effect (Ref 8:2707).

GDOS

The chemical composition of the material sputtered into the glow discharge plasma is representative of that of the substrate. The light emitted from the discharge contains emission lines corresponding to the atomic species of that substrate. The intensity of the impurity spectral line is proportional to the concentration of impurity atoms. This relationship can be obtained by first calibrating the GDOS system using pure standards. The calibration procedure will be explained in Chapter VI of this report.

Thus, it has been shown that GDOS sputtering is accomplished in an abnormal glow discharge which is characterized by the glow completely covering the cathode. Light emitted from the cathode glow region of the discharge contains emission lines corresponding to the atomic species of that substrate. After calibrating the system, the intensity of the impurity spectral line can be related to the concentration of the impurity atoms. The experimental apparatus used in this study is presented in Chapter IV.

IV. Experimental Apparatus

The equipment used for this study has been reported in a previous thesis study (Ref 23:18-26). The equipment modifications accomplished in the present work are described in Chapter V of this report. For the convenience of the reader, this chapter will contain a brief overview of the GDOS equipment. A schematic view of the GDOS system used in these experiments is shown in Figure 5 (Ref 23:19). A dual trace XY recorder has been substituted for the single trace recorder used previously. Analyzing both profiles on one recorder provides better depth resolution of the impurity profile. The other single trace recorder was used for accurate concentration determination because of its expanded scale.

Sputtering Chamber

The heart of the GDOS system is the pyrex glass sputtering chamber shown in Figure 6 (Ref 23:20). Ultra-violet light is transmitted through the silica quartz windows to the substrate and impurity detection systems. The anode is constructed of a 1½" diameter cylindrical stub made of stainless steel. The cathode is a hollow electrode fitted with inlet and outlet tubes for circulation of cooling nitrogen gas. The cathode surface is actually a tungsten (W) plate cemented on an aluminum cap.

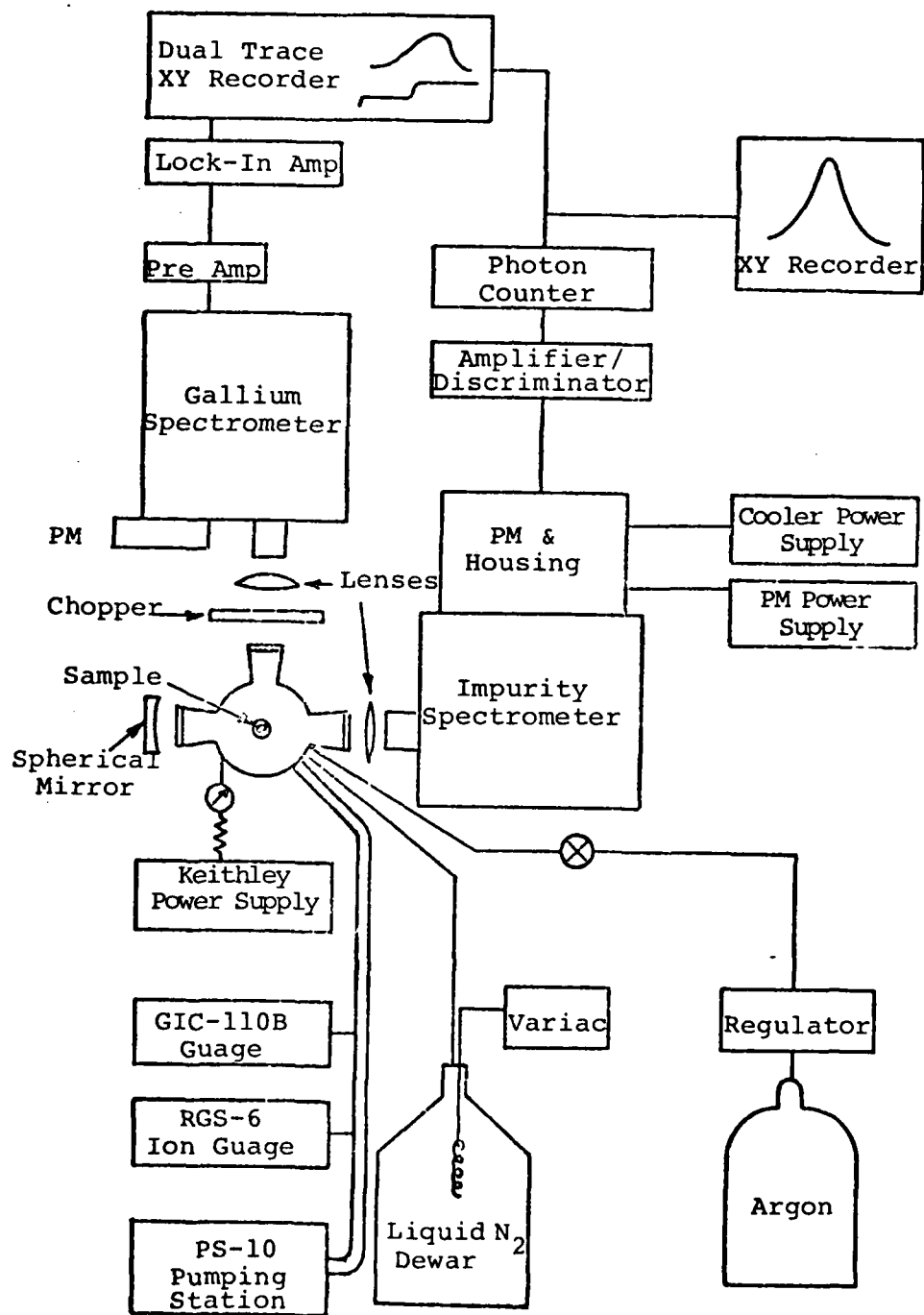


Figure 5. GDOS Apparatus (From Ref 23:19)

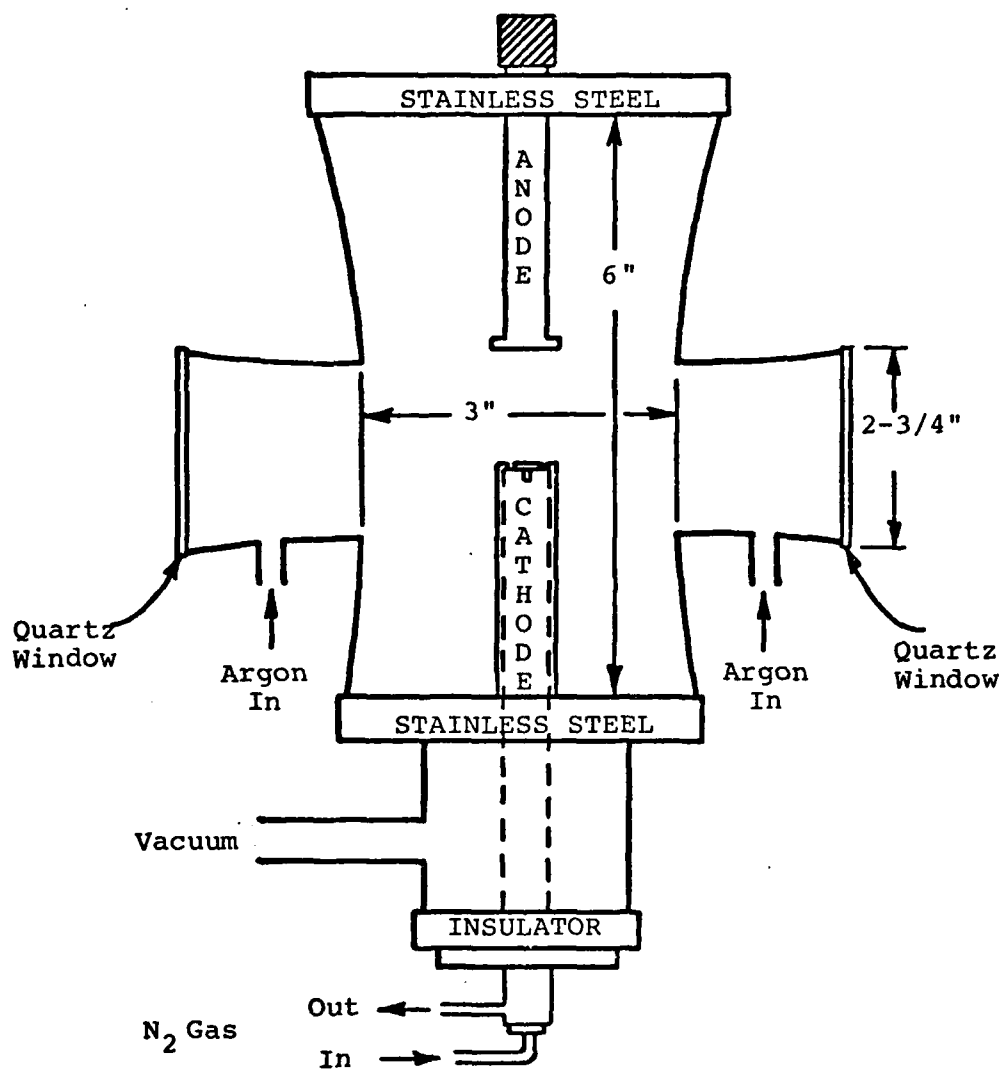


Figure 6. Sputtering Chamber

The cap and electrode are protected from being sputtered by an insulating pyrex shield as shown in Figure 7 (Ref 23:22). The cathode to anode separation is variable and the voltage across them can range to ± 3500 volts.

Vacuum, Gas, and Cooling System

The chamber is evacuated using the PS-10 pumping station consisting of a mechanical pump for roughing and for backing of the diffusion pump, and an oil diffusion pump with a liquid nitrogen cooled trap for high vacuum. Regulated Ar gas is supplied to the chamber through a leak valve and the cooling nitrogen gas for the cathode is provided by a ten liter Dewar container with a submersed heating element controlled by a Variac autotransformer.

Detection and Recording System

Detection of the impurity and substrate emission is accomplished by using two independent systems. One system monitors the impurity line and the other system monitors the Ga in the substrate.

Light gathered from the impurity line is focused on the entrance slit of a one-meter Czerny-Turner spectrometer (Refer to Figure 5). Light reflects off the diffraction grating and is channeled through the exit slit to a thermoelectrically cooled photomultiplier tube. The output of this tube is passed through an Amplifier/Discriminator into a photon counter where the photon counts are

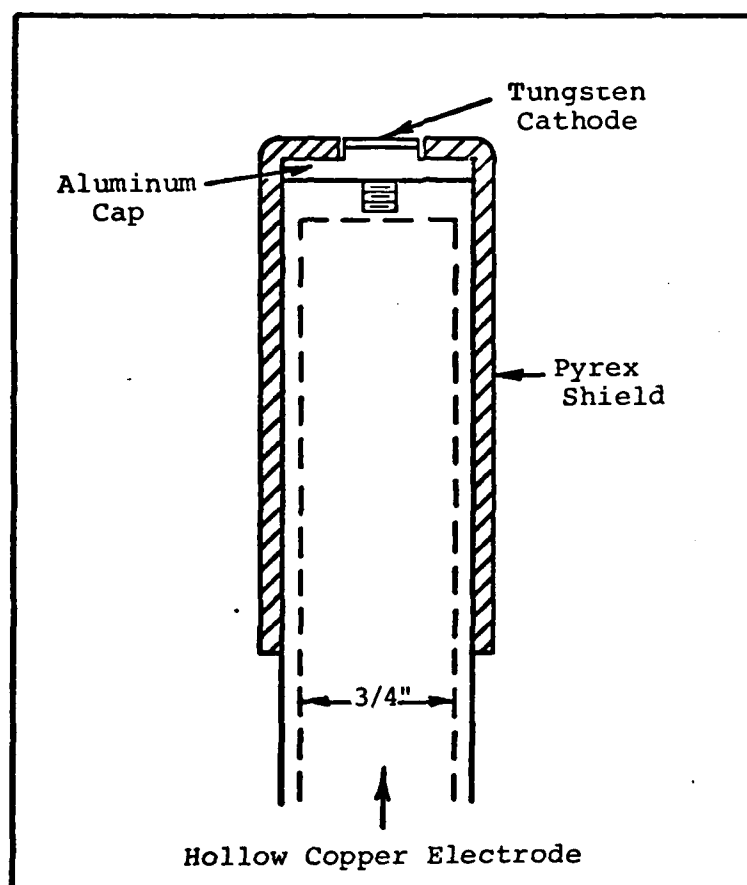


Figure 7. Cathode Configuration (From Ref 23:22)

displayed. The logarithmic voltage output of the photon counter is then displayed on dual trace and single trace XY recorders.

Light gathered from the Ga line is chopped and then focused on the entrance slit of a 3/4 meter Czerny-Turner spectrometer (refer to Figure 5). After reflecting off the diffraction grating, the light is passed through the exit slit onto a photomultiplier tube. The output from the photomultiplier tube is amplified and then passed through a lock-in amplifier to enhance the S/N ratio. Signal output is then displayed on the dual trace XY recorder.

The basic equipment used in this study of GDOS profiling has now been defined. The apparatus consists of a sputtering chamber, the vacuum gas and cooling systems, and the detection and recording system. Detection is accomplished by two separate systems: one for the impurity line of interest, and one for the Ga line. Together these two detection systems provide the information necessary for impurity profiling in GaAs. To optimize the capabilities of the system, improvements to the vacuum system and sputtering chamber were accomplished. This is discussed in Chapter V of this report.

V. System Improvements

Electrical, Vacuum, and Gas Improvements

As stated earlier in this report, a considerable amount of time was devoted to optimizing the optical system and improving the vacuum integrity. This optimizing included repair of major components and acquisition of replacements for defective equipment. Complete overhaul of both the diffusion and mechanical pumps, along with a systematic leak repair procedure enhanced the efficiency of the vacuum system. Upon completion, evacuation to 2×10^{-5} torr could be accomplished in 30 minutes as compared to previous times of two to three hours (Ref 25). Now the system could be set up and a sample profiled in less than two hours whereas the same profiling would take between 4 and 5 hours previously. Thus, the productivity of the GDOS system was doubled.

Included in this vacuum system repair was a replacement of the argon line, bypass, and leak valve to the chamber. A single line was run from the Ar regulator to a new leak-bypass system (Figure 8). This change decreased the leak valve to chamber distance from 8½ feet to approximately 6 inches. This allowed faster system response to the Ar inputs and also less outgassing and less volume for evacuation.



Figure 8. Leak-Bypass Valve System on Vertical Chamber

Sputtering Chamber Improvements

In the abnormal glow discharge, the entire cathode is covered by the cathode glow. The chamber used previously was configured such that the glow discharge and detection windows were at right angles to each other. This geometry as shown in Figure 8 shows that only a small portion of the cathode glow is viewed and collected using this arrangement. One of the premises of this study was that decreasing the angle between the discharge and detection window would increase the amount of light collected since only a small solid angle of light is intercepted when the window is 90° from the sample. Detection at 60° was selected for optimum viewing and collecting of light emitted from the cathode. Detection at smaller angles resulted in the anode itself blocking some of the radiance.

Two chambers, one made of glass and the other aluminum, were built with modified detection windows. Although the aluminum chamber could be machined more precisely than the glass chamber, the welds around the arms of the chamber leaked, and a good vacuum could not be achieved, thus it was not utilized.

The modified glass chamber was installed after new metal bracing was built to hold the chamber at 30° from horizontal. The chamber and bracings are shown in Figure 9.

With the chamber held in this position, the W cathode needed to be redesigned in order to keep the substrate chip from falling off. This was accomplished by

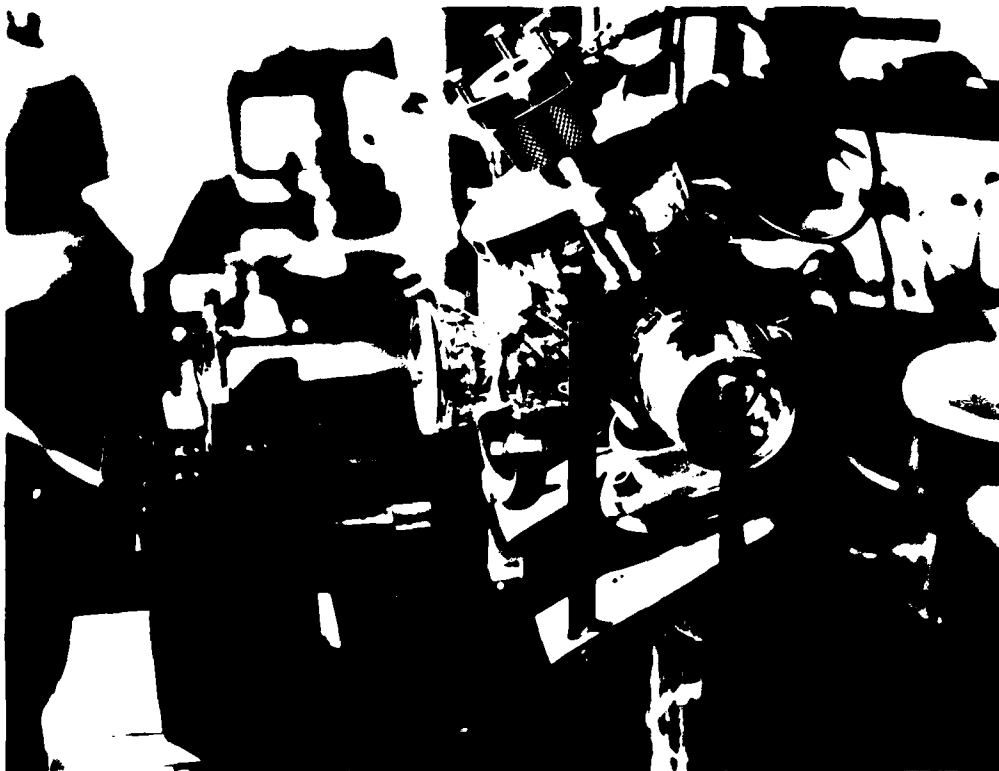


Figure 9. Modified Chamber Arrangement

taking a 40 mil thick W sheet and cutting out a one inch circular disk. This disk was then ground to make a 20 mil deep step to hold the chip. The step was designed such that the GaAs chip was centered on the W after placement of the peripheral chips. The peripheral chips were used to enhance uniform surface sputtering of the substrate since they limit access of the electric field lines to the sides of the sample. Sharp edges cause high local current densities and strong electric fields near them, leading to increased sputtering at the edges. A top view of the cathode with the substrate and peripheral chips mounted is shown in Figure 10.

Improvements on Light Collection

Sensitivity of the GDOS system is a function of the light gathering capability of the optics and the signal handling capability of the electronics. Increasing the efficiency of the optics should increase the sensitivity of the system by lowering the threshold of the concentration that can be observed.

Single Lens System. For impurity light collection, previous GDOS studies have used a plano-convex lens to image the source onto the entrance slit of the spectrometer. This lens was secured to the spectrometer using a bar clamp and lens holder. A new system was designed and built during this study which allowed the lens to be translated in

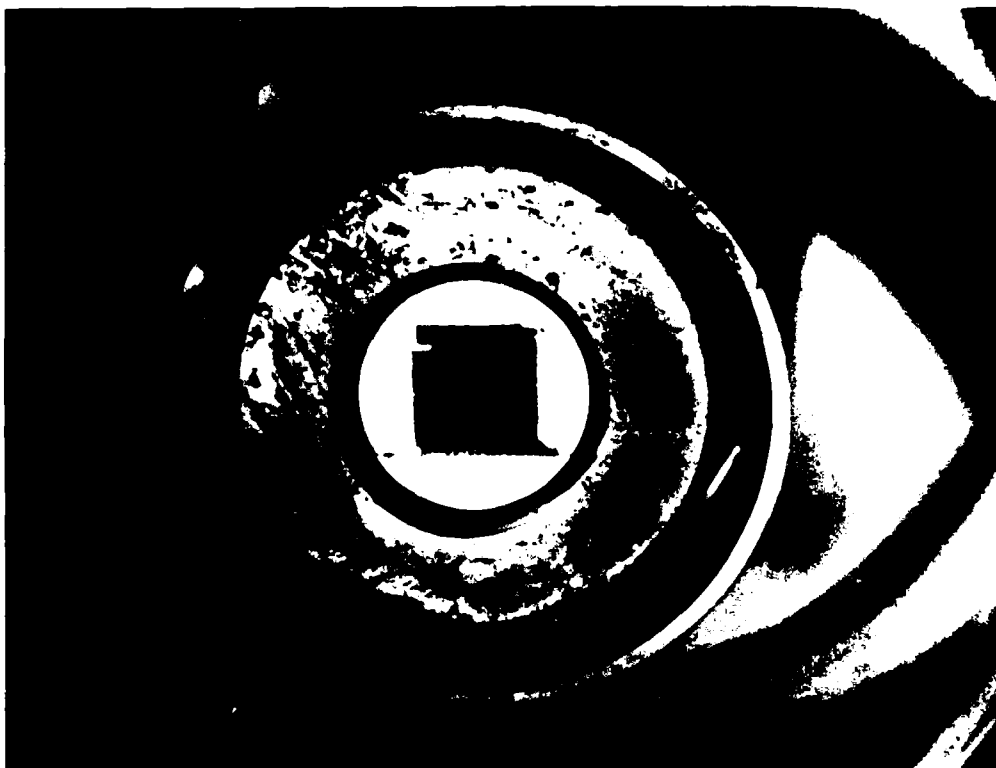


Figure 10. Sample and Peripheral Chips Mounted on Modified Tungsten Chamber

three directions thereby making lens adjustments easier and reducing the noise induced by lens vibrations.

The lens focal length was previously reported to be 6.5 cm and the lens was placed at 2f or 13 cm from the spectrometer and substrate (Ref 26). A lens placed at 2f from an object will image at 2f distance from the lens according to the Gaussian lens formula:

$$\frac{1}{S_o} + \frac{1}{S_i} = \frac{1}{f} \quad (1)$$

where

S_o = object distance,

S_i = image distance, and

f = focal length of the lens.

The actual focal length of this lens was measured on an optical bench to be 10.0 cm, requiring a 20.0 cm distance from the 2f stipulation. Correcting this distance increased the amount of light passing through the spectrometer for two reasons. First, the image of the light emitted by the glow is now focused on the entrance slit. The previous lens arrangement provided a focused image 43.3 cm from the lens and consequently focused light did not enter the spectrometer.

The second reason changing the lens to spectrometer distance increased the radiance gathered by the spectrometer is simply that the f-numbers of the lens and spectrometer

were better matched. The f-number ($f/\#$) is defined as follows:

$$f/\# \equiv \frac{X}{D} \quad (2)$$

where

X = focal length of monochromator or the image distance of the lens, and

D = the diameter of the diffraction grating or the diameter of the lens.

Maximum light collection will occur when the f-numbers of the lens and spectrometer are matched as shown in Figure 11. The term Ω is usually referred to as the solid angle. If Ω' is less than Ω , the grating will not be fully illuminated and the throughput will be reduced. If Ω' is equal to Ω , maximum throughput is achieved.

The f-number of the Czerny-Turner one meter spectrometer is $f/9$. Assuming the diameter of the lens to be 5 cm, the f-number was increased from $f/2.6$ to $f/4$ by increasing the distances S_o and S_i to 20.0 cm. When the amount of light entering a 200 micron wide entrance slit is compared for each of these two cases, the result is an increase of 3.5 times higher radiance input.

One inherent problem in this experimental setup is the loss of light due to the size of the image. The object, or cathode glow, is a flat horizontal pancake shaped spot which when imaged will also be flat, horizontal and pancake

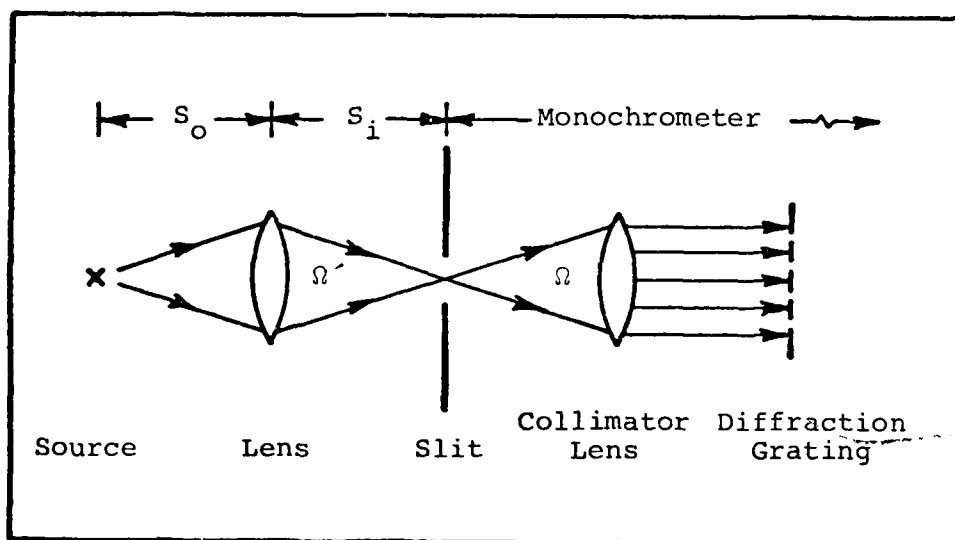


Figure 11. f/# Matching

shaped. The vertical spectrometer input slit only allows a portion of the image in.

One solution to this would be to rotate the slit and diffraction grating. The spectrometer used in this experiment can only accommodate such changes by tilting the entire spectrometer on its side. With the spectrometer on its side, and the entrance slit set at 200 microns, radiance input would increase by a factor of 10 since the entire image would fall within the entrance slit. Another possible solution is to reduce the size of the image. The Luminous Conservation Law states that a reduced image will have more irradiance (flux/area) than the same size image, but the solid angle will increase proportionally. But increased solid angle can only be accomplished by locating the lens closer to the spectrometer, thus reducing S_i . Decreasing S_i decreases the f-number of the lens system which reduces the amount of light into the spectrometer. The best trade-off between f-number matching and transverse magnification is accomplished when the lens is set at the $2f$ distance from the object and image plane, and the transverse magnification of object to image is one-to-one.

Two Lens System. Better matching of the f-number of the spectrometer can be accomplished using a two lens system. Using the principles that light passing through a lens focal point and incident on that lens will exit the lens in parallel rays, and that parallel light incident

on another lens will exit through that lens focal point, an optimal system can be devised as shown in Figure 12. The distance from the source to lens A and from lens B to the slit must be equal to the focal lengths of lenses A and B respectively. The distance from lens A to lens B has little effect on the analysis.

Using equation (2) and knowing the spectrometer f-number ($f/9$) and the diameter of the secondary lens (5 cm), f-number matching can be accomplished if lens B has a focal length of 45 cm. The focal length of lens A can be found by using the magnification criterion. That is, that the image should be the same size or smaller than the object. The magnification of this system is calculated by dividing the focal lengths of the input lenses (f_B/f_A). Thus, the focal length of lens A must be 45 cm or greater.

Surface Losses. As light passes through a glass-air interface, a reflectance loss of approximately 4% is introduced (Ref 9:80). A two lens system will introduce four interfaces and therefore only 84.9% of the light incident on the first lens will pass through the second lens. The remaining light is lost due to reflection. A single lens system will pass 92.2% of the incident light since there are only two interfaces. Coating the lens with an anti-reflection coating reduces the 4% reflectance to approximately 1% at each interface (Ref 9:314). With these coatings applied, a single lens system will pass 98% of the incident light and a two lens system will pass 96%.

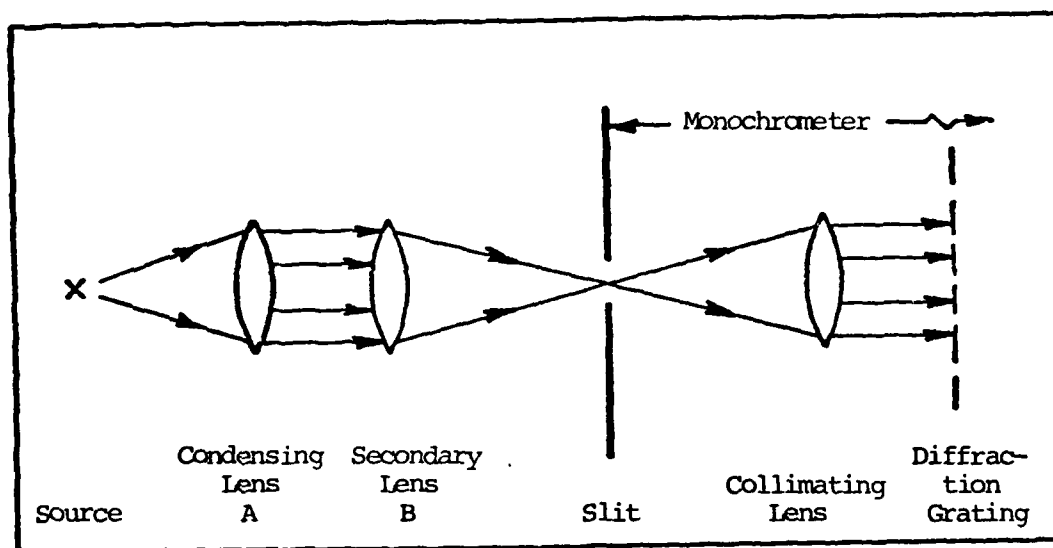


Figure 12. Two Lens f/# Matching

Rearward Light Collection. In previous studies, a 3 inch diameter spherical mirror was placed directly opposite the collecting lens to enhance the light collecting efficiency of the system by collecting and sending forward light emitted in the rearward direction. Since a mirror has the same imaging properties as a lens except that the object and image are always both on the same side, the mirror was placed a distance of twice its focal length from the cathode. However, no increased light collection was noticed (Ref 26).

A spherical mirror set of $2f$ distance from an object will produce an inverted same size image at $2f$ distance. Since the light collected is from slightly above the cathode, the inverted image of the light was focused on the cathode itself. This blocked most of the light from being passed forward. Placing the mirror at a distance of f from the cathode also proved ineffective. Light from the cathode will reflect in parallel rays off the mirror. This light is then collected and focused by the forward lens. However, the forward lens is at a $2f$ distance from the entrance slit of the spectrometer. The parallel light incident on this lens is focused at the f distance from the lens and is diverging rapidly when it reaches the spectrometer. The overall effect is that little additional light enters the slit of the spectrometer.

Enhancement of the GDOS system light collection could be accomplished by replacing the concave mirror with

a planar mirror and lens. With this lens placed at its focal length distance from the cathode, light from the cathode will be emitted parallel from the rear line, reflect off the mirror, and be focused back onto the cathode. The light will now be passed forward to be collected and focused on the spectrometer entrance slit (Figure 13).

Mirror Losses. Optical quality mirrors are usually constructed by a process of evaporating and depositing a metal film onto a smooth clean surface. The reflectivities of these metals are highly wavelength dependent. Ag and Al are normally used because of their ease of evaporation and their highly reflective nature. Ag mirrors will reflect a maximum of 96% at wavelengths of 3800\AA and higher. Al also has a flat response peaked at 90% for wavelengths of 2400\AA and higher. Below 3800\AA and 2400\AA the reflectivity curves for Ag and Al drop sharply (Ref 17:1066).

Previous studies of this GDOS apparatus have used Ag mirrors for reflection of the rearward light. The reflection of an Ag mirror for the Ge line at 3037\AA is a maximum of 10%. Although Al mirrors provide a maximum of only 90% reflection, the response is flat at 90% to wavelengths well below 3037\AA for Fe. Depending on the wavelength of the impurity being profiled, Al mirrors can be used with a slightly better response than Ag mirrors.

Optimum Optical System. The conclusion of the foregoing analysis is that the optimum optical system would be

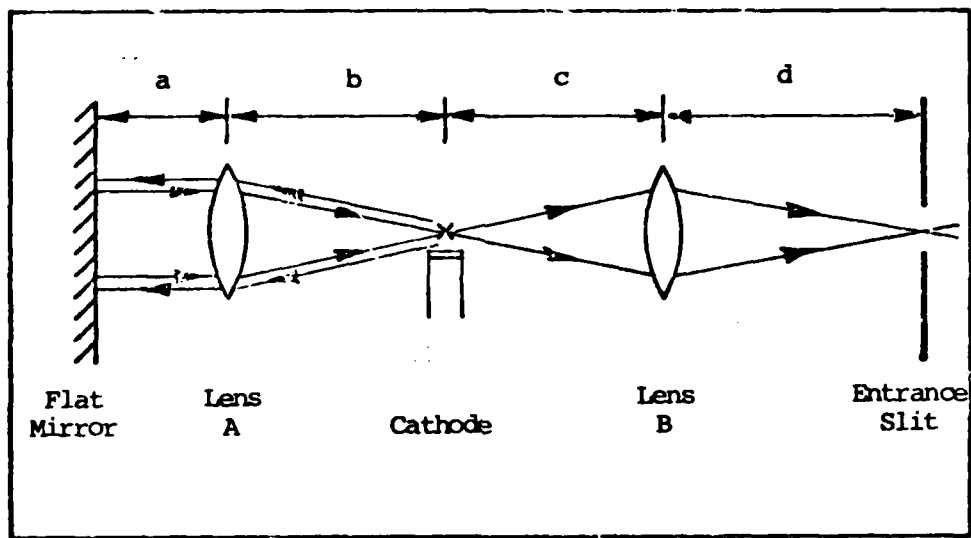


Figure 13. Optimum Optical System

one designed as in Figure 13. The focal length of lens A and B should be 45 cm and 22.5 cm respectively. Distance a can vary since it merely measures the distance the parallel light will travel. Distances b, c, and d should be 22.5 cm, 45 cm and 45 cm. Both lenses should be coated with an antireflection coating and the mirror should be a flat Al coated mirror. These improvements should more than double the light collection capability of the present GDOS system.

The many improvements made to the GDOS equipment in this study were made in an effort to increase the efficiency of the system. An analysis of various lens and mirror arrangements was conducted in an effort to find an optimum optical system. This system when installed should increase the efficiency of the GDOS light collection capabilities. Utilizing many of the foregoing improvements, the experimental procedures used in this investigation are presented in Chapter VI.

VI. Experimental Procedure

Sample Preparation

The samples used in this experiment were cut from wafers of melt grown, undoped GaAs prepared by Crystal Specialties Laboratories. Each sample was cut square 0.5 cm on each side. The samples were surrounded by perimeter chips which eliminate both the high local current densities and increased sputtering on the sharp edges of the substrate. The perimeter chips, shown previously in Figure 10, were also cut from undoped GaAs substrate material and were 0.125 cm by 0.6 cm.

Before implantation, all samples were cleaned using a standard cleaning procedure involving a sequence of chemical rinses. Surface contaminants must be removed prior to ion implantation or these contaminants will be driven into the crystal during the implantation procedure. Ion implantation was performed using a highly modified 150 keV accelerator manufactured by Accelerators, Inc. of Austin, Texas. Ge ions were implanted at 120 keV at a fluence of 5×10^{14} ions/cm² and 10^{15} ions/cm² and at 90 keV at a fluence of 10^{15} ions/cm². All samples were implanted at room temperature and were mounted 8° from the normal for amorphous implantation. After implantation, the samples were cleaned and then capped with 1000Å of Si₃N₄. This cap, formed in a plasma of silane and nitrogen

at a temperature of 300°C, is called a plasma enhanced Si_3N_4 cap. The purpose of this cap was to permit the discharge to stabilize prior to sputtering the implanted substrate.

Sputtering Rate Determination

In order to find the profile of the implanted impurity ions, the sputtering rate of the substrate must first be determined. This was accomplished by placing a small chip on top of the sample being sputtered. When the discharge was turned on, the portion of the substrate covered by the chip was left unsputtered and thus a step in thickness was produced between sputtered and unsputtered regions (Figure 14). This step was measured using a Sloan Dektak Surface Measuring System consisting of the Dektak and associated chart recorder. Dividing the amount of sputtered substrate by the measured time period gives a sample sputtering rate determination. The sputtering rate was also determined for the plasma enhanced Si_3N_4 cap. The results are given in Chapter VII.

Since the cathode was at 30° for most of this study, an alternate method had to be devised to hold the top chip on the substrate. This was accomplished using a 0.2 cm long top chip and another peripheral chip cut thick enough (0.08 cm by 0.75 cm) to be set on its side to hold the top chip. This is shown pictorially in Figure 10. The substrate and the top chip are both 0.05 cm thick, so

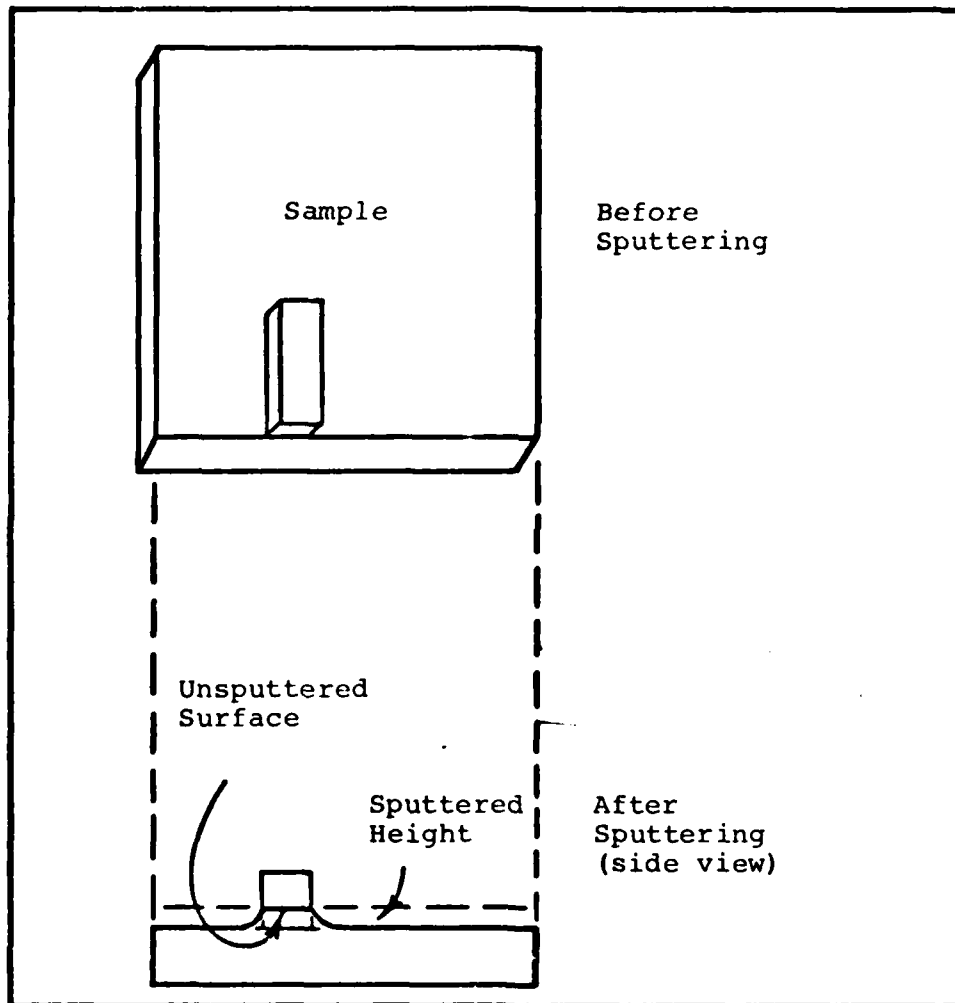


Figure 14. Sputtering Rate Determination

a 0.08 cm wide chip set on its side adequately holds the top chip from falling off the sample.

System Operation

A sample was first placed in the sputtering chamber and the chamber was evacuated to 2×10^{-5} torr. During the evacuation, nitrogen gas was flowed through the cathode for cooling. After evacuation was complete, argon gas was admitted to the chamber through a leak valve in order to maintain $20 \pm 1/2$ microns of pressure. After the pressure was stabilized, the recorders were turned on and the discharge was started by applying 2100 volts across the electrodes. The discharge requires a few seconds to attain a steady-state sputtering condition. During this time surface contaminants are removed by the sputtering (Ref 2:618).

System Calibration

Calibration of the GDOS system was accomplished using samples of pure Ge and Mg. First the spectrometer was tuned to a strong characteristic line of the substrate to be detected. The concentration of impurity ions in an implanted sample was determined by linearly relating the calculated density of the pure elements to the observed intensity of their spectral line. This relationship was based on the assumption that implanted atoms ionize as readily as atoms of the pure element. This assumption was experimentally shown to be valid for Ge and Mg as discussed in the next chapter. The accuracy of this calibration

technique has been proven by previous comparisons of profiles obtained by GDOS to profiles using AES and SIMS (Ref 7).

Impurity Profiles

Impurity concentration profiles were measured by tuning the spectrometer to a strong emission line of the particular impurity while the sample was being sputtered. The location of the best spectral lines for several elements of interest are tabulated in spectral tables (Ref 18). The spectral line which had the strongest intensity and least interference from other spectral lines was selected. To be sure no interfering lines were close enough in wavelength to deposit energy into the impurity line of interest, spectra were recorded for unimplanted GaAs, a pure sample of the dopant (e.g., a pure Ge sample) and a GaAs sample doped with the impurity. The recorded spectra from these three runs were superimposed and the exact location and strength of the impurity line and its proximity to other spectral lines were determined.

For accurate profile analysis, depth measurements must be taken from the surface of the substrate. To do so, the exact location of the cap-surface interface must be determined. By monitoring the intensity of the Ga line of the GaAs substrate, the sudden increase in Ga signal can be used to indicate the location of the surface (Figure 15). The time to this point can be determined

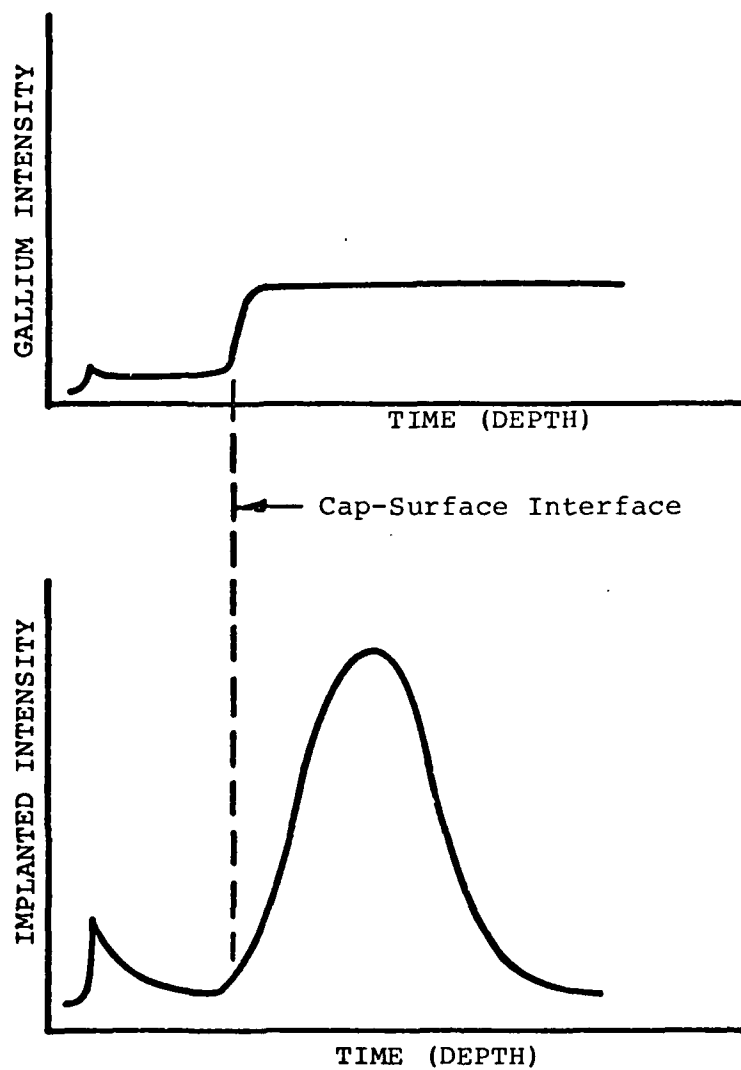


Figure 15. Determination of Cap-Surface Interface (Ref 18:38)

and the depth of the impurity peak is measured from this point.

The profiles were calculated by plotting the intensity of the implanted spectral line as a function of time and comparing the known sputtering rate and time of sputtering to determine depth measurements. The result is a plot of concentration versus depth into the substrate.

The experimental procedures for operating the GDOS system and plotting the impurity profiles obtained have now been discussed. The depth of the impurity peak can be determined if the sputtering rate of the implanted GaAs and the exact cap-surface interface is known. The concentration of the impurity can be determined by linearly relating the observed intensity of the impurity spectral line to the calculated density of the pure element. The results of these experiments are discussed in Chapter VII.

VII. Results

Experimental data obtained in this study were compared to that obtained in a successful previous GDOS thesis study (Ref 22). Emphasis was placed on comparing the two chamber arrangements, vertical and modified. The modified chamber was installed and the front lens was arranged such that there was a $2f$ distance from cathode to lens and lens to spectrometer. The tilted chamber blocks all light from being emitted horizontally backwards, and so no mirror was used behind the chamber. After sputtering many samples at different chamber pressures and voltages, it was determined that the most even sputtering across the entire sample was achieved with a voltage of 2100 volts and a chamber pressure of 20 microns. This pressure and voltage were used for all subsequent samples described in this report.

Calibration Results

Sputtering rates were determined for unimplanted GaAs as well as samples implanted with Ge. The sputtering rate was measured at $1400\text{\AA}/\text{min}$ for both cases. This can be explained by the fact that the impurity concentrations are small and, as a result, the sputtering rate is determined entirely by the GaAs substrate. The sputtering rate

for Si_3N_4 was determined to be 525 \AA /min. These rates were used in determining depth measurements in all experiments.

Light passing through the input slit of the one-meter spectrometer is focused on a photomultiplier tube which responds to the number of photons incident on the detector by amplifying this signal and producing an electrical output signal. This signal is digitized and sent to a photon counter which displays the number of photon counts present during each second. Thus, the intensity of input light is proportional to the number of count observed. The sensitivity of the GDOS system is measured by how many sputtered atoms/cc are represented by one count of the photon counter. This sensitivity measurement was analyzed for Ge and Mg in this report.

The observed intensity of sputtered Ge was found to be 102,000 counts at 1400 \AA /min sputtering rate. Linearly scaling down the sputtering rate to 1150 \AA /min results in approximately 83,800 counts of Ge. This linear scaling is necessary to compare the results of this experiment to previous studies. The atomic number density of Ge is calculated by the following equation:

$$\frac{1}{72.59 \text{ AMU}} \cdot \frac{1 \text{ AMU}}{1.659 \times 10^{-24} \text{ gm}} \cdot \frac{5.323 \text{ gm}}{\text{cc}} = 4.417 \times 10^{22} \text{ atoms/cc}$$

The proportionality constant between observed intensity and concentration for Ge is:

$$4.417 \times 10^{22} \text{ atoms/cc} \frac{1}{83,800 \text{ counts}} = 5.27 \times 10^{17} \text{ atoms/cc/count}$$

Williamson found that with a sputtering rate of 1150Å/min, he obtained 1×10^{18} atoms/cc/count (Ref 23:34). This indicates that the light collecting efficiency of the system has been increased by a factor of two by redesigning the cathode and chamber.

Williamson proved that the relationship between observed intensity and concentration was truly linear by reducing the size of the sample being sputtered and noting that the intensity reduced proportionally (Ref 23:34). The same experiment was repeated in this study with the same results.

A similar analysis was performed using Mg. The observed intensity of the sputtered Mg was 930,000 counts at a sputtering rate of 1400Å/min. Scaling this down to 1150Å/min sputtering rate results in approximately 763,900 counts of sputtered Mg. Using the same method as above, the atomic number density of Mg is 4.31×10^{22} atoms/cc. The proportionality constant between observed intensity and concentration of Mg is now 5.6×10^{16} atoms/cc/count as compared to previous results of 1.02×10^{17} atoms/cc/count (Ref 23:35). Again, this shows an increase in efficiency by a factor of two.

The minimum impurity detection limit is an important quantity to know since it can be directly related to

a minimum ion dose or fluence which can be detected. Given an ion dose, the peak LSS concentration can be calculated by the following equation (Ref 6:1-10):

$$N_{MAX} = \frac{\phi}{\sqrt{2\pi} \overline{\Delta R}_p} \quad (3)$$

where

N_{MAX} = peak LSS concentration (atoms/cc),

ϕ = ion dose (atoms/cm²), and

$\overline{\Delta R}_p$ = standard deviation of projected range.

Dividing N_{MAX} by the proportionality constant (in atoms/cc/count) obtained for a specific element, yields the amount of counts the GDOS system will display at the projected range depth. Conversely, assuming that there must be at least 90 counts of signal to be distinguishable from the noise, the minimum ion dose can be calculated by reworking these equations to find ϕ . Previously, the minimum detectable ion dose for Ge at 120 keV implantation energy was 5×10^{14} /cm². Using the present proportionality constant of 5.27×10^{17} atoms/cc/count, the minimum detectable fluence is now 3×10^{14} /cm².

Helium Addition

Helium studies were performed on a pure Ge substrate. The number of counts of sputtered Ge at 1400Å/min was 102,000 counts before the introduction of He. With 5% He introduced, this reading decreased to 76,000 counts.

The number of counts did not increase past 102,000 until the amount of He was reduced to 1%, at which time the intensity increased to 108,000 counts. The reading was also approximately 108,000 counts at 0.5% He. The sputtering rate was determined to be approximately 1400Å/min for the Ge with 1% He introduced. Thus, the amount of collected light increased by 6% with no appreciable increase in sputtering rate when less than 1% of He was introduced into the plasma.

While performing the above experiment, it was found that the He lingers in the vacuum system. To insure that the amount of He remaining in the chamber did not affect the next measurements, the chamber was evacuated to 5×10^{-5} torr after each run.

Germanium Implants

Impurity profiles were obtained for the Ge-implanted GaAs samples. The measured profiles show the impurity depth to be within 5% of the LSS calculated projected range. The measured projected ranges for the 90 keV and 120 keV implantation energies were 338Å and 467Å respectively. The LSS projected depths of Ge for these energies are 355Å and 476Å (Ref 6).

The measured peak concentrations were obtained by multiplying the number of observed Ge counts by the proportionality constant for Ge. At 90 keV energy and at a fluence of 10^{15} ions/cm², the measured peak of 2.1×10^{20} /cm³

is comparable to the LSS predicted peak of $2.3 \times 10^{20}/\text{cm}^3$. At 120 keV, the measured peak concentrations for fluences of 5×10^{14} ions/ cm^2 and 10^{15} ions/ cm^2 were $8.9 \times 10^{19}/\text{cm}^3$ and $1.7 \times 10^{20}/\text{cm}^3$ which parallel the LSS values of $9.1 \times 10^{19}/\text{cm}^3$ and $1.8 \times 10^{20}/\text{cm}^3$ respectively. An example profile is shown in Figure 16.

Chromium Profiling

Upon sputtering a pure Cr sample, high intensity spectral lines were observed corresponding to Cr emission lines. Spectra were taken of pure Cr, GaAs:Cr, and undoped GaAs. All three spectra showed the same Cr lines but at different intensities. After removing the sample and sputtering the W cathode alone and observing a strong Cr spectral line, it was determined that there was Cr in the chamber. Upon further investigation it was determined that the stainless steel anode is made up of 16 to 20% Cr and that the tungsten cathode had some Cr impurities in it. The conclusion is that Cr can only be profiled in the present GDOS system if the cathode surface and the entire anode are replaced with a metal containing no Cr.

Vertical Chamber

A replica of the original vertical chamber was built and experiments were run using this chamber. Without using any rear optics, the Ge forward light collection was determined to be 103,000 counts. This is approximately

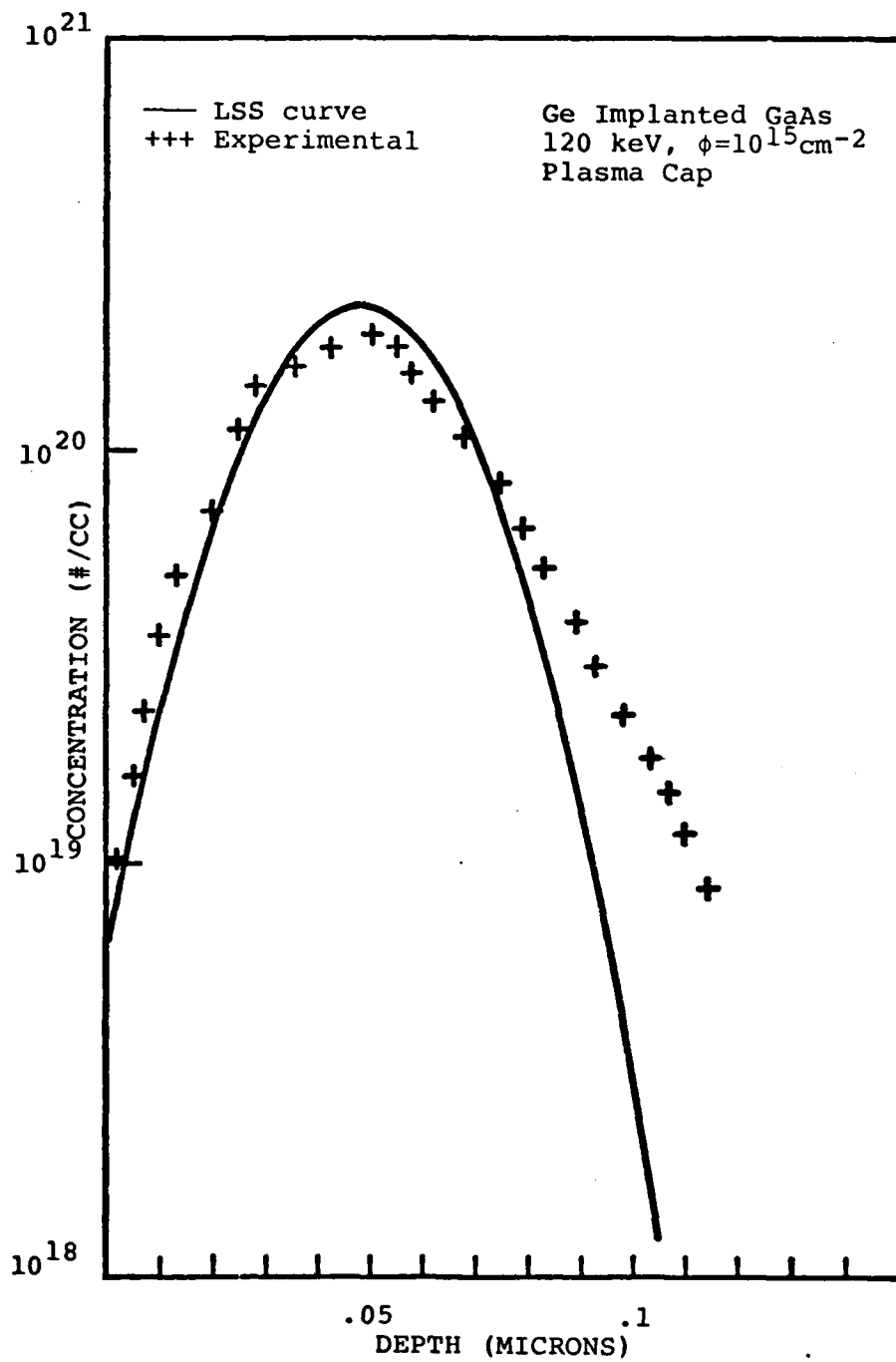


Figure 16. Measured GDOS Profile of Ge-Implanted GaAs Compared to Theoretical LSS Profile

the same amount of light that was collected from the tilted chamber, which indicates that the tilted chamber arrangement produced no additional light collection, but that the improvements made to the lens system had increased the sensitivity of the GDOS system by a factor of two.

To insure that the results obtained were accurate, exhaustive testing was performed to insure even sputtering across the sample. With this chamber installed, 2000 volts at 22 microns of pressure was required to obtain even sputtering. This produced a $1400\text{\AA}/\text{min}$ sputtering rate of unimplanted GaAs and all experiments using this chamber were performed using this voltage and pressure.

In order to collect the rearward light, a mirror was fabricated by evaporating and depositing a thin film of Al onto a 4 inch diameter flat glass plate. The mirror was clouded during the evaporation process which reduced its reflectivity from 90% to less than 70%. A 3 cm diameter quartz lens with a focal length of 15 cm was placed behind the chamber at 15 cm f distance from the cathode. The mirror was placed behind this lens. The result was an increase in light collection of a sputtered Ge sample from 103,000 counts to 153,000 counts which further reduced the minimum impurity detection limit to 3.51×10^{17} atoms/cc/count. This was verified by sputtering some Ge-implanted GaAs samples. Again, the depths and concentrations of Ge atoms observed paralleled the LSS predicted values. Helium was introduced into this chamber

resulting in 6% increased light collection for 1% He introduced. With He introduced in this chamber and lens arrangement, the minimum distinguishable ion dose is calculated to be $2 \times 10^{14}/\text{cm}^2$.

Thus, the modified chamber arrangement was investigated and the results indicate that the threshold limit for Ge detection was lowered from 1×10^{18} atoms/cc/count to 5.27×10^{17} atoms/cc/count. With He introduced into the chamber, the plasma efficiency was increased by 6% and this further lowered the threshold to 4.97×10^{17} atoms/cc/count. Impurity profiles were obtained for Ge-implanted GaAs samples. The measured projected ranges and peak concentrations paralleled the LSS predicted values. Cr profiling was not performed due to strong interfering Cr spectral lines produced by Cr in the chamber anode and cathode.

The vertical chamber was installed and compared to the tilted chamber. The minimum impurity detection limit was found to be identical. This indicated that improvements in the lens system made during this study had increased the sensitivity of the GDOS system by a factor of two. The tilted chamber produced no additional sensitivity increase, proving that the light emitted in the glow discharge is emitted randomly and thus there is no difference in the intensity of the light collected from the solid angle of light emitted horizontally or at 30° . A quartz

lens and a Al coated mirror was placed behind the vertical chamber. Observed Ge counts increased to 153,000 counts which further reduced the minimum detection limit to 3.51×10^{17} atoms/cc/count. With 1% He introduced into the chamber, the amount of collected light was increased by 6%. This reduced the minimum to its present operating limit of 3.32×10^{17} atoms/cc/count. Thus, the present system is 3.0 times more efficient than the GDOS system used in previous studies and the minimum distinguishable ion dose of Ge at 120 keV implantation is now $2 \times 10^{14}/\text{cm}^2$. Conclusions based on the results of this investigation are presented in Chapter VIII.

VIII. Conclusions and Recommendations

Conclusions

The following conclusions can be drawn concerning the use of the GDOS technique for total impurity profiling of ion implanted GaAs:

1. The improved vacuum integrity and Ar gas regulation has provided a more responsive, accurate and sensitive GDOS system. Evacuation to 2×10^{-5} torr could now be accomplished in less than 30 minutes as compared to previous times of two to three hours. A complete profiling procedure can be accomplished in less than two hours as compared to 4 to 5 hours previously. This effectively doubled the productivity of the GDOS system.
2. It was determined that the tilted chamber did not increase the sensitivity of the GDOS system. The factor of two increase in system sensitivity was attributed to improvements made to the lens system. A further improvement was noticed by introducing small amounts of He (less than 1%) into the chamber. This increased the light collection efficiency of the system by approximately 6%.
3. Cr profiling in the present GDOS system is not possible due to the Cr impurities present in the cathode and anode metals. Refabricating the cathode and anode of a metal containing no Cr impurities is necessary before Cr profiling can be performed.

4. The optimum chamber arrangement is the vertical chamber with an Al coated flat mirror and silica quartz lens to capture light emitted in the rearward direction and forward light collected by a single quartz lens. The minimum impurity detection limit of the present GDOS system was found to be 3.32×10^{17} atoms/cc/count, which is 3.0 times more efficient than the GDOS system used in previous studies. By using the optimum lens arrangement as discussed in this report, with all lenses coated with antireflection coatings and with He introduced, the minimum impurity detection limit should decrease to approximately 2×10^{17} atoms/cc/count. By placing the spectrometer on its side, the radiance input will increase by a factor of 10 and the detection limit will further decrease to 2×10^{16} atoms/cc/count.

5. The most important limitation of the GDOS system is the requirement that the substrate be implanted with very high doping concentrations. The previous minimum detection limit of 10^{18} atoms/cc/count translates into a minimum detectable ion dose for Ge of $5 \times 10^{14}/\text{cm}^2$. The more sensitive present system has reduced the detectable fluence to $2 \times 10^{14}/\text{cm}^2$. If the optimum arrangement suggested in this study is implemented complete with coated lenses and the spectrometer set on its side, the projected 10^{16} atoms/cc/count minimum detection limit would translate into a minimum detectable ion dose of $10^{13}/\text{cm}^2$. Although this doping is still very high, there may be some

applications for heavily doped PN junction devices where the GDOS system would be useful. An example would be ohmic contacts which are usually $10^{13} - 10^{14}$ atoms/cm².

Recommendations

This investigation has expanded previous studies of the GDOS profiling technique. The following recommendations are made to point out possible uses for the GDOS system:

1. An attempt should be made to implement the optimum GDOS system presented in this study. This should include addition of He gas, AR coated lenses, a flat Al mirror, and the one meter spectrometer set on its side. After these modifications have been made, the minimum detection limit of 10^{16} atoms/cc/count should be attainable.
2. The cathode surface and the entire anode should be replaced with a metal containing no Cr impurities. Profiling studies of Cr in GaAs:Cr should be possible since the amount of bulk doped Cr ranges from 10^{16} to 10^{17} atoms/cc. This concentration is well within the detection capabilities of the GDOS system.
3. The GDOS system should be used for residual impurity examination. Detection of Mn, a primary cause of thermal conversion in GaAs, should be possible. Other residual impurities could be detected, providing an inexpensive in-house evaluation procedure for the ion implantation process.

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Vita

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The glow discharge optical spectroscopy (GDOS) technique was investigated to determine if the sensitivity of the system could be improved. GDOS was applied to determine impurity concentration profiles of annealed and unannealed ion implanted GaAs samples. Modifications were made to the sputtering chamber and the light collection systems which resulted in a 3.7 times increase in system sensitivity. Comparisons were made to previous studies by sputtering GaAs samples implanted with Ge at energies of 90 keV and 120 keV and fluences of		

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$5 \times 10^{14}/\text{cm}^2$ and $10^{15}/\text{cm}^2$. The implanted samples were sputtered in a low pressure argon gas discharge. Intensity of a strong emission line, characteristic of the implanted impurity, was monitored as a function of time. Profiles of Ge implanted GaAs were measured and analyzed.

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